Joint Oil Analysis Program
Spectrometer Standards
SCP Science (Conostan) Qualification Report
For D19-0, D3-100, and D12-XXX Series Standards

NF&LCFT REPORT 441/15-008

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EXECUTIVE SUMMARY

The Joint Oil Analysis Program previously revised the specification for their spectrometric oil standards in order to incorporate an ICP-AES test method and to transition to commercially manufactured spectrometric oil standards. Historically, a Rotrode-AES test method was the only elemental test method used to verify the quality of the spectrometric oil standards. The Rotrode-AES test method was a labor and time intensive process that did not exhibit a high level of accuracy and repeatability. The JOAP wanted to incorporate an ICP-AES test method in order to obtain a more accurate determination of the true concentration of the elements in the spectrometric oil standards and to reduce the cost and time needed to conduct quality assurance. Both the Rotrode-AES and ICP-AES test methods are now part of the qualification inspection in order to ensure that commercially manufactured spectrometric oil standards are identical to those that have been produced in-house by the JOAP.

This report covers the qualification testing of Type D19-0, Type D12-XXX series, and Type D3-100, candidate spectrometric oil standards submitted by SCP Science (Conostan) against the requirements of MIL-DTL-85694. In general, the candidate spectrometric oil standards met the requirements of the specification. A few specific data points were analyzed and waived based on alternative testing and analysis. As a result of this testing and analysis effort the SCP Science (Conostan) Type D19-0, Type D12-XXX series, and Type D3-100 spectrometric oil standards have been qualified to the specification.

Since this qualification testing effort was only the second time the full range of data required by MIL-DTL-85694 has been collected, analysis of the data has led to the conclusion that several of the requirements of the specification need revision. Several changes to the specification are therefore under consideration based on historical knowledge of JOAP manufactured spectrometric oil standards and the MIL-DTL-85694 qualification data and analysis from all of the commercially manufactured spectrometric oil standards.

LIST OF ACRONYMS/ABBREVIATIONS

Joint Oil Analysis Program ..... JOAP

Joint Oil Analysis Program-Executive Committee ..... JOAP-EC

Rotrode Atomic Emission Spectroscopy ..... Rotrode-AES

Inductively Coupled Plasma Atomic Emission Spectroscopy ..... ICP-AES

Joint Oil Analysis Program Spectrometer Standards SCP Science (Conostan) Qualification Report For D19-0, D3-100, and D12-XXX Series Standards

1.0 BACKGROUND

The Joint Oil Analysis Program, reference (a), previously revised the specification for their spectrometric oil standards in order to incorporate an ICP-AES test method and to transition to commercially manufactured spectrometric oil standards. Historically a Rotrode-AES test method was the only elemental test method used to verify the quality of the spectrometric oil standards. The Rotrode-AES test method was a labor and time intensive process that did not exhibit a high level of accuracy and repeatability. The JOAP wanted to incorporate an ICP-AES test method in order to obtain a more accurate determination of the true concentration of the elements in the spectrometric oil standards and to reduce the cost and time needed to conduct quality assurance. Both the Rotrode-AES and ICP-AES test methods are now part of the qualification inspection in order to ensure that commercially manufactured spectrometric oil standards are identical to those that have been produced in-house by the JOAP.

SCP Science (Conostan) submitted Type D19-0, Type D12-XXX series, and Type D3-100 spectrometric oil standards that were manufactured to meet the requirements of MIL-DTL-85694. Testing was accomplished at the NAVAIR Patuxent River MD test facility in accordance with the latest revision of MIL-DTL-85694, reference (b).

2.0 OBJECTIVE

The objective of this testing is to qualify a second commercial vendor, SCP Science (Conostan), for the production of blended spectrometric oil standards that are used in calibrating and verifying the calibration of spectrometers used in spectrometric analysis of metallic elements found in oils and other fluids.

3.0 APPROACH

The test methods for qualification inspection are specified in Table I. The density, viscosity, viscosity index, trace sediment, pour point, and flash point test methods were conducted in accordance with the corresponding ASTM test method listed in Table I.

TABLE I. Test Methods

Physical property	Test Method
Density at 60 °F	ASTM D4052
Viscosity (mm^2 /s at 100 °C)	ASTM D445
Viscosity Index, Min	ASTM D2270
Trace Sediment	ASTM D2273
Pour point	ASTM D97
Flash point, minimum (COC)	ASTM D92
Metal Elements (Rotrode-AES)	ASTM D6595
Metal Elements (ICP-AES)	ASTM D5185

The Rotrode-AES test method was conducted in accordance with ASTM D6595. Testing of candidate spectrometric oil standards was done as a statistical analysis and performed on the data generated by the spectrometric analysis to provide an accuracy index and repeatability index for each element involved. The results were obtained with a JOAP-EC approved Spectroil M/N spectrometer (S/N 6403). The testing consisted of ten replicates of each candidate spectrometric standard.

The ICP-AES test method was conducted in accordance with ASTM D5185. Testing of spectrometric oil standards was done as a statistical analysis and performed on the data generated by the spectrometric analysis to provide an accuracy index for each element involved. The results were obtained with a JOAP-EC approved ICP spectrometer (Agilent 725 Series ICP-OES spectrometer). The ICP spectrometer was calibrated from 0 ppm to 20 ppm using a commercial ICP multi-element spectrometric standard. Candidate spectrometric oil standards were diluted on a weight-by-weight basis with a suitable ICP solvent. Candidate spectrometric oil standards in concentrations 5 ppm, 10 ppm, 30 ppm, and 50 ppm were diluted tenfold while candidate standards in concentrations 80 ppm, 100 ppm, 120 ppm, 300 ppm, 500 ppm, 700 ppm, and 900 ppm were diluted to 5 ppm. The testing consisted of five replicates of each candidate spectrometric standard.

4.0 DISCUSSION

This section details the analysis of the data in accordance with MIL-DTL-85694 qualification testing. In the situations where a requirement was not met, the NOAP-EC reviewed the data and determined if the specific requirement can be waived due to factors that influenced some chemical and physical properties that were not taken into consideration during the initial development of the specification.

4.1 Type D19-0

The Type D19-0 spectrometric oil standard submitted by the candidate met the requirements for density at 60°F, viscosity at 100°C, viscosity index, trace sediment, and flash point (Table II).

TABLE II. Candidate Type D19-0 Physical Property Results

TEST	METHOD	UNITS	SAMPLES
			D19-0
Density at 60°F	ASTM D4052	kg/L	0.8837
Viscosity at 100°C	ASTM D445	cSt	18.5
Viscosity @ 40°C	ASTM D445	cSt	211.9
Viscosity Index	ASTM D2270	No Units	96
Pour Point	ASTM D97	°C	**-11**
Flash Point	ASTM D92	°C	255
Water & Sediment	ASTM D2273	%	0.000

NOTE: Red numbers indicate the requirement not being met

The D19-0 did not meet the requirement for Pour Point however the measured value is within the reproducibility for ASTM D97 and the NOAP-EC is willing to waive the requirement.

The Type D19-0 spectrometric oil standard submitted by the candidate met the requirement for Rotrode-AES (Table III) and ICP-AES (Table IV).

TABLE III. Candidate Type D19-0 Rotrode-AES Results

TEST	METHOD	UNITS	SAMPLES	
			D19-0	
Trace Metal:	ASTM D6595	ppm	Mean	Stdev
Al			*0.0*	*0.0*
Cr			*0.0*	*0.0*
Cu			*0.0*	*0.0*
Fe			*0.1*	*0.2*
Pb			*0.1*	*0.1*
Mg			*0.0*	*0.0*
Ni			*0.0*	*0.0*
Si			*0.0*	*0.0*
Ag			*0.0*	*0.0*
Na			*0.0*	*0.0*
Sn			*0.4*	*0.3*
Ti			*0.0*	*0.0*
B			*0.0*	*0.0*
Mo			*0.0*	*0.0*
Zn			*0.5*	*0.0*
Ba			*0.0*	*0.0*
Cd			*0.0*	*0.0*
Mn			*0.3*	*0.2*
V			*0.1*	*0.2*

TABLE IV. Candidate Type D19-0 ICP AES Results

TEST	METHOD	UNITS	SAMPLES
			D19-0
ICP	ASTM D5185	ppm	Mean
Al			*0.0*
Cr			*0.0*
Cu			*0.1*
Fe			*0.0*
Pb			*-0.1*
Mg			*0.1*
Ni			*-0.3*
Si			*-0.6*
Ag			*0.0*
Na			*0.1*
Sn			*0.0*
Ti			*0.0*
B			*0.1*
Mo			*0.0*
Zn			*0.4*
Ba			*0.0*
Cd			*0.0*
Mn			*-0.1*
V			*-0.2*

The Type D19-0 spectrometric oil standard submitted by the candidate satisfactorily passed the qualification inspection.

4.2 Type D12-XXX Series

All of the Type D12-XXX spectrometric oil standards submitted by the candidate met the requirements for density at 60°F, viscosity at 100°C, viscosity index, and trace sediment (Table V).

TABLE V. Candidate Type D12-XXX Physical Property Results

TEST	METHOD	UNITS	SAMPLES					
			D12-5	D12-10	D12-30	D12-50	D12-100	D12-300
Density at 60°F	ASTM D4052	kg/L	0.8813	0.8847	0.8704	0.8859	0.8867	0.8918
Viscosity at 100°C	ASTM D445	cSt	18.5	18.5	18.6	18.6	18.7	19.0
Viscosity @ 40°C	ASTM D445	cSt	212.3	212.6	213	213.2	213.7	214.6
Viscosity Index	ASTM D2270	No Units	96	96	97	97	97	99
Pour Point	ASTM D97	°C	-14	-14	-14	-14	**-11**	-15
Flash Point	ASTM D92	°C	**228**	**237**	255	262	263	**211**
Water & Sediment	ASTM D2273	%	0.000	0.000	0.000	0.000	0.000	0.000

NOTE: Red numbers indicate the requirement not being met

The D12-30 and D12-50 spectrometric oil standards met the physical property requirements while the D12-5, D12-10, and D12-300 oil standards met all of the physical property requirements except for flash point and the D12-100 oil standard met all of the physical property requirements except for pour point.

The D12-100 did not meet the requirement for Pour Point; however the measured value is within the reproducibility for ASTM D97 and the NOAP-EC is willing to waive the requirement.

The D12-5 and D12-10 did not meet the requirement for Flash Point; however the measured value is within the reproducibility for ASTM D92 and the NOAP-EC is willing to waive the requirement.

The high concentration of the metallo-organic concentrates in D12-300 has an effect on the flash point which resulted in the requirement for flash point not being met. The significance of the effect was not taken into consideration during the development of the original specification requirements therefore the flash point requirement for D12-300 will require revision. At this time the NOAP-EC is waiving the flash point requirement for D12-300.

All of the Type D12-XXX spectrometric oil standards with the exception of D12-300 met the requirement for Rotrode-AES (Table VI). The Rotrode-AES chromium, iron, and nickel accuracies for D12-300 were identified as being outside of the Rotrode-AES requirement. Following standard JOAP quality assurance procedure, the D12-300 was diluted to 50 ppm using D19-0 and retested to address the accuracy issue. Upon retesting, the Rotrode-AES accuracy and repeatability requirements for all elements were met (Table VII).

All of the Type D12-XXX spectrometric oil standards met the requirement for ICP-AES (Table VIII).

TABLE VI. Candidate Type D12-XXX Rotrode-AES Results

TEST	METHOD	UNITS	SAMPLES											
			D12-5		D12-10		D12-30		D12-50		D12-100		D12-300	
Trace Metal:	ASTM D6595	ppm	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev
Al			*4.9*	*0.2*	*9.7*	*0.2*	*29.6*	*0.7*	*47.4*	*1.0*	*100.3*	*2.2*	*330.6*	*8.8*
Cr			*5.5*	*0.2*	*11.0*	*0.2*	*30.1*	*0.7*	*51.8*	*2.3*	*101.3*	*2.8*	***341.2***	*10.5*
Cu			*5.2*	*0.2*	*10.5*	*0.2*	*31.0*	*0.7*	*51.4*	*1.5*	*103.8*	*3.5*	*315.4*	*14.4*
Fe			*5.1*	*0.2*	*10.7*	*0.2*	*31.3*	*0.9*	*52.8*	*1.8*	*110.5*	*3.0*	***361.6***	*12.9*
Pb			*5.4*	*0.5*	*11.6*	*0.2*	*31.8*	*1.0*	*53.7*	*1.3*	*106.3*	*1.7*	*321.2*	*9.0*
Mg			*5.0*	*0.3*	*10.4*	*0.2*	*30.8*	*0.6*	*51.0*	*1.2*	*102.5*	*2.1*	*292.7*	*8.9*
Ni			*4.9*	*0.2*	*10.1*	*0.2*	*30.0*	*0.5*	*50.6*	*1.1*	*100.9*	*1.8*	***339.7***	*10.4*
Si			*5.0*	*0.3*	*10.5*	*0.2*	*30.4*	*0.9*	*51.1*	*1.4*	*101.6*	*2.3*	*320.9*	*9.7*
Ag			*4.9*	*0.2*	*9.5*	*0.2*	*30.4*	*0.7*	*48.8*	*1.4*	*102.5*	*3.4*	*282.2*	*9.5*
Na			*5.3*	*0.1*	*10.6*	*0.3*	*30.3*	*1.0*	*51.7*	*1.8*	*99.7*	*3.3*	*279.4*	*8.4*
Sn			*4.9*	*0.7*	*10.3*	*0.5*	*30.5*	*1.0*	*50.7*	*1.5*	*99.0*	*1.3*	*297.7*	*5.2*
Ti			*4.5*	*0.2*	*9.5*	*0.3*	*29.2*	*0.8*	*50.4*	*2.4*	*103.9*	*2.1*	*317.1*	*5.9*

NOTE: Red numbers indicate the requirement not being met

TABLE VII. Retest of Candidate Type D12-XXX Rotrode-AES Results

TEST	METHOD	UNITS	SAMPLES											
			D12-5		D12-10		D12-30		D12-50		D12-100		D12-300	
Trace Metal:	ASTM D6595	ppm	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev	Mean	Stdev
Al													*46.3*	*0.7*
Cr													*46.1*	*0.6*
Cu													*48.5*	*0.9*
Fe													*49.7*	*1.1*
Pb													*50.0*	*0.9*
Mg													*49.0*	*0.8*
Ni													*48.4*	*0.7*
Si													*49.0*	*0.7*
Ag													*44.8*	*0.9*
Na													*50.8*	*1.8*
Sn													*46.9*	*0.7*
Ti													*47.3*	*1.1*

TABLE VIII. Candidate Type D12-XXX ICP-AES Results

TEST	METHOD	UNITS	SAMPLES					
			D12-5	D12-10	D12-30	D12-50	D12-100	D12-300
ICP	ASTM D5185	ppm	Mean	Mean	Mean	Mean	Mean	Mean
Al			*5.0*	*9.9*	*29.5*	*49.0*	*97.7*	*292.6*
Cr			*5.2*	*10.4*	*31.2*	*54.6*	*102.6*	*308.8*
Cu			*5.0*	*10.0*	*29.8*	*49.9*	*99.0*	*297.1*
Fe			*5.1*	*10.2*	*30.5*	*50.4*	*100.8*	*302.8*
Pb			*5.0*	*9.9*	*29.7*	*50.2*	*99.6*	*298.9*
Mg			*5.2*	*10.4*	*31.1*	*51.1*	*102.5*	*306.9*
Ni			*5.0*	*10.3*	*31.1*	*51.7*	*103.8*	*313.4*
Si			*4.4*	*9.2*	*29.2*	*49.0*	*98.1*	*297.7*
Ag			*5.0*	*10.1*	*30.3*	*50.3*	*100.0*	*300.1*
Na			*4.8*	*9.6*	*28.9*	*48.6*	*96.5*	*289.9*
Sn			*5.0*	*9.9*	*30.2*	*50.3*	*100.4*	*301.6*
Ti			*4.6*	*9.3*	*27.8*	*46.4*	*93.0*	*279.4*

The Type D12-XXX spectrometric oil standards submitted by the candidate satisfactorily passed the qualification inspection.

4.3 Type D3-100

The Type D3-100 spectrometric oil standard submitted by the candidate met the requirements for density at 60°F, viscosity at 100°C, viscosity index, trace sediment, pour point, Rotrode-AES, and ICP-AES (Table IX).

TABLE IX. Candidate Type D3-100 Physical Property, Rotrode AES and ICP AES Results

TEST	METHOD	UNITS	SAMPLE	
			D3-100	
Density at 60°F	ASTM D4052	kg/L	0.8854	
Viscosity at 100°C	ASTM D445	cSt	18.5	
Viscosity @ 40°C	ASTM D445	cSt	221.2	
Viscosity Index	ASTM D2270	No Units	93	
Pour Point	ASTM D97	°C	-14	
Flash Point	ASTM D92	°C	**222**	
Water & Sediment	ASTM D2273	%	0.000	
Trace Metal:	ASTM D6595	ppm	Mean	Stdev
B			*98.429*	*3.325*
Mo			*107.3*	*7.371*
Zn			*94.84*	*3.822*
ICP	ASTM D5185	ppm	Mean	
B			*118.8*	
Mo			*86.4*	
Zn			*99.9*	

NOTE: Red numbers indicate the requirement not being met

The D3-100 did not meet the requirement for Flash Point and is not within the reproducibility for ASTM D92; however the NOAP-EC is willing to waive the requirement due to the fact that current JOAP spectrometric standards have flash points as low as 110°C.

The Type D3-100 spectrometric oil standard submitted by the candidate satisfactorily passed the qualification inspection.

5.0 CONCLUSIONS

The following spectrometric oil standards submitted by SCP Science (Conostan) have satisfactorily passed the qualification inspection and should be added to the qualified products list for MIL-DTL-85694:

- Type D19-0
- Type D12-XXX (D12-5, D12-10, D12-30, D12-50, D12-100, D12-300)
- Type D3-100

6.0 RECOMMENDATIONS

A number of issues associated with the specification requirements were brought to light as a result of all of the qualification testing. Several changes to the specification are therefore under consideration.

Commercial manufacturers have notified the NOAP-EC that they are having difficulty obtaining SAE J1966, reference (c), oil due to low market demand. The NOAP-EC and commercial manufacturers have been informed that SAE J1899, reference (d) could be an acceptable alternative to SAE J1966 as long as the oil is silicon free however manufacturers are having difficulty obtaining silicon free J1899 for the same reason. Changes to MIL-DTL-85694 are therefore under consideration in order to better define chemical and physical property requirements and help address the challenges associated with supply.

Density and viscosity are the key physical properties that could have significant influence on Rotrode measurements. Flash point can be most influenced by the metallo-organic concentrates and stabilizer. The base oil has less of an effect on flash point. Current MIL-DTL-85694 JOAP spectrometric oil standards have flash points as low as 100°C therefore flash point is a less critical physical property requirement.

The pour point test method has a large reproducibility range. In addition, pour point is influenced by the chemical composition of the oil. The challenges that commercial manufacturers are having associated with supply due to market demand could explain a variation in the chemical composition of the oil and thus the higher pour point. Pour point did not appear to adversely influence Rotrode-AES measurements and therefore is a less critical physical property requirement.

The NAVAIR Patuxent River MD test facility has recently expanded its test capability by adding instrumentation able to conduct viscosity measurements in accordance with ASTM D7042. For this reason, ASTM D7042 is under consideration for inclusion in the next revision of MIL-DTL-85694, reference (b).

7.0 REFERENCES

a. OPNAVINST 4731.2, Joint Oil Analysis Program, 26 March 2014
b. MIL-DTL-85694A, Detail Specification, Spectrometric Oil Standards, 12 August 2013
c. SAE J1966, Lubricating Oil, Aircraft Piston Engine (non-Dispersant Mineral Oil), REV. JUL2005
d. SAE J1899, Lubricating Oil, Aircraft Piston Engine (Ashless-Dispersant), REV. AUG2005

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Appendix A
Viscosity Test Data

The NAVAIR Patuxent River MD test facility has recently expanded its test capability by adding instrumentation able to conduct viscosity measurements in accordance with ASTM D7042. Viscosity data collected in accordance with ASTM D7042 are presented in Table X in addition to the corresponding viscosity index data.

TABLE X. ASTM D7042 Viscosity Results and Corresponding ASTM D2270 Viscosity Index Results

TEST	METHOD	UNITS	SAMPLES							
			D19-0	D12-5	D12-10	D12-30	D12-50	D12-100	D12-300	D3-100
Viscosity at 100°C	ASTM D7042	cSt	18.5	18.5	18.5	18.6	18.6	18.7	19.0	18.6
Viscosity @ 40°C	ASTM D7042	cSt	212.9	213.1	213.6	214.1	214.3	214.8	215.8	214.46
Viscosity Index	ASTM D2270	No Units	96	96	96	96	97	97	99	96

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14. ABSTRACT

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